

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF:

NORIFUMI SUMIMOTO ET AL

SERIAL NO. 09/729,927 GROUP ART UNIT: 1714

EXAMINER: TAE H. YOON

FOR: FLAME RETARDANT THERMOPLASTIC RESIN COMPOSITION

DECLARATION UNDER 37 C.F.R. 1.132

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WASHINGTON, D.C. 20231

SIR:

Now comes Norifumi SUMIMOTO, a citizen of Japan, and a resident of c/o Techno Polymer Co., Ltd., 18-1, Kyobashi 1-chome, Chuo-ku, Tokyo, Japan, who declares and says that:

1. I graduated from Shizuoka University, Faculty of Engineering (master degree), in March, 1989.
2. I was employee of JSR Corporation in 1989-1996, I have been an employee of Techno Polymer Co., Ltd since 1996 and have been engaged in the study of an ABS resin composition.
3. I am an inventor of U.S. Patent Application, Serial No. 09/729,927.
4. I have read the Office Action dated May 22, 2003, have understood the Examiner's rejection of the invention claimed

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in the above application. Then, under my control, the following experiments were conducted.

Experiments 1 and 2

(1) Preparation of rubber polymer:

As rubber polymer (a-1), there was used polybutadiene latexes shown in Table 1 below.

Table 1

Rubber polymer	(a-1)
Polybutadiene latex	
Particle size distribution (%)	
not more than 150nm	12
from more than 150 nm to less than 350 nm	80
not less than 350 nm	8
Gel fraction (%)	78

(2) Preparation of component (A):

The rubber polymer (a-1) was emulsion-polymerized with styrene and acrylonitrile as the monomer component (b) at mixing ratios shown in Table 2 thereby obtaining graft copolymer (A1-1). Separately, only styrene and acrylonitrile as the monomer component (b) were solution-polymerized with each other at mixing ratios shown in Table 2, thereby obtaining copolymer (A2-1). The intrinsic viscosities  $[\eta]$  of the thus obtained graft copolymers (A1) and copolymers (A2) are shown in Table 2.

Table 2

	Rubber polymer		Monomer component (part)		Graft ratio (%)	Intrinsic viscosity [η] (dl/g)
	Kind	Part	Styrene	Acrylonitrile		
A1-1	(a-1)	30	49	21	115	-
A2-1	-	-	70	30	-	0.56

(3) Preparation of phosphorus-based flame retardant:

The following condensed phosphoric acid esters (B-1) to (B-2) were used as the component (B).

(B-1): Condensed phosphoric acid ester represented by the above general formula (I) wherein R<sup>1</sup> to R<sup>4</sup> are phenyl; X is a residue of bisphenol A; and n is 1.1.

(B-2): Condensed phosphoric acid ester represented by the above general formula (I) wherein R<sup>1</sup> to R<sup>4</sup> are phenyl; X is a residue of bisphenol A; and n is 1.4.

(4) Preparation of component (C):

As the component (C), there was used ethylene bis-stearylamine produced by Kao Co., Ltd.

(5) Preparation of resin composition:

The respective components were mixed together for 3 minutes at mixing ratios shown in Table 3 using a Henschel mixer. Then, the resultant mixture was melt-extruded from an NVC-type 50-mm vented extruder manufactured by Nakatani Kikai Co., Ltd., by setting the cylinder temperature to 180 to 220°C,

thereby obtaining pellets. The thus obtained pellets were sufficiently dried, and then injection-molded using an injection molding machine J100E-C5 manufactured by Nippon Seikosho Co., Ltd., by setting the cylinder temperature and mold temperature to 200°C and 50°C, respectively, thereby obtaining test specimens for various evaluation tests. The test specimens were tested by the following evaluation methods.

Particle size and particle size distribution of rubber polymer:

The sizes of particles dispersed in latex were measured by laser Doppler/frequency analysis. The measurement was conducted using a granulometer ("MICRO-TRACK UPA150, MODEL NO. 9340" manufactured by Nikkiso Co., Ltd.). Meanwhile, it was confirmed that the size of the rubber polymer particles dispersed in the rubber-reinforced resin were substantially identical to those dispersed in latex.

Gel fraction (content of toluene-insoluble components):

The gel fraction was measured by the above method described in the present specification.

Graft ratio (percentage):

The graft ratio was measured by the above method described in the present specification.

Intrinsic viscosity  $[\eta]$ :

The rubber-reinforced resin was added into acetone. The resultant mixture was shaken at room temperature for 2 hours using a shaker, and then centrifuged for 60 minutes using a centrifugal separator (rotating speed: 23,000 rpm), thereby

separating the mixture into acetone-insoluble components and acetone-soluble components. The obtained acetone-soluble components were sufficiently dried by a vacuum dryer. The dried acetone-soluble components were dissolved in methyl ethyl ketone to prepare five solutions having different concentrations. The reduced viscosities of the five solutions was measured at 30°C by Ubbellode viscometer. The intrinsic viscosities  $[\eta]$  (unit: dl/g) was calculated from the thus measured viscosities.

Fluidity (melt flow rate):

The melt flow rate (unit: g/10 minutes) was measured at 220°C under a load of 98N according to JIS K7210.

Impact resistance (Izod impact strength):

A test specimen No. 2 according to JIS K7110 was molded using an injection molding machine J100E-C5 manufactured by Nippon Seikosho Co., Ltd., by setting cylinder temperature and mold temperature thereof to 220°C and 50°C, respectively. The Izod impact strength (unit: J/m) of the test specimen was measured according to ASTM D256.

Heat deformation temperature (HDT):

A test specimen having a size of 6.4 mm in width x 128 mm in length x 12.8 mm in thickness, was measured under a bending stress of 18.5 kgf/cm<sup>2</sup> according to JIS K7207.

Flammability evaluation (flame retardancy):

A test specimen of 5" in length x 1/2" in width x 1/12" in thickness was subjected to vertical flame test by the method prescribed in UL94. In the evaluation results, "V-2"

represents V-2 acceptance in the vertical flame test, and "B" represents "burning", i.e., V-2 non-acceptance.

Falling weight impact strength:

The breaking energy of a test specimen having a size of 50 mm x 80 mm x 2.4 mm was measured using a high-speed impact tester "SERVO-PULSER EHF-2H-20L" manufactured by Shimadzu Seisakusho Co., Ltd. The measuring conditions were as follows: Specimen pedestal diameter: 30mm $\phi$ ; Striking bar tip: 12.7mmR; Striking speed: 3.1 m/s. The unit of the falling weight impact strength is "kgf·cm".

The results are shown in Table 3.

Table 3

Experiment	1	2
Composition (part)		
Component (A)		
(A1-1)	40	40
(A2-1)	60	60
Properties of component (A)		
Rubber content (%)	12	12
Graft ratio (%)	115	115
Component (B)		
(B-1) (n=1.1)	10	-
(B-2) (n=1.4)	-	10
Component (C)	2	2
Evaluation results		
Fluidity (g/10 min.)	49	20
Izod impact strength (J/m)	14	8
Heat deformation temperature (HDT) (°C)	83	85
Burning property	V-2	V-2
Falling weight impact strength	390	120